Alfred Goldberg

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THE EFFECT OF CONCURRENT STRAINING ON THE ANNEALING BEHAVIOR OF A COLD-ROLLED VACUUM-MELTED ELECTROLYTIC IRON

BY

ALFRED GOLDBERG AND SUMNER GURNEY

American Iron and Steel Institute
150 East Forty-Second Street
New York 17, N.Y.

Attention: Mr. Charles M. Parker, Vice President Research and Technology

Dear Sir:

Attached hereto is a technical report which has been prepared for distribution. The effect of polygonization on modifying the annealing behavior of a cold-rolled steel subjected to creep was pointed out in a previous report. In order to examine more closely the effects of concurrent straining during annealing the present investigation was undertaken using a purer metal in which polygonization should occur more readily. Instead of creep, constant strain rate loading techniques were employed.

The cooperation and support given by the American Iron and Steel Institute in making these studies possible are sincerely appreciated. The authors also would like to acknowledge the interest shown by the U.S. Naval Postgraduate School and the Office of Naval Research in supporting the development of the laboratory facilities for this general area of research.

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THE EFFECT OF CONCURRENT STRAINING ON THE ANNEALING BEHAVIOR OF A COLD-ROLLED VACUUM-MELTED ELECTROLYTIC IRON

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ALFRED GOLDBERG[1] AND SUMNER GURNEY[2]

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ABSTRACT

The annealing behavior of a cold-rolled iron was investigated with and without the presence of concurrent straining. The analyses are based on hardness and microstructural observations.

In the absence of concurrent straining a temperature range was encountered where polygonization and recrystallization were highly competitive resulting in considerable scatter in the hardness data. This occurred only on annealing at the lower temperatures.

The effect of concurrent straining was studied at a slow and fast strain rate at two temperatures. An immediate hardness drop, independent of the test variables, was observed. This was attributed to the elimination of vacancy clusters during the initial straining. At the lower temperature with additional concurrent straining softening was accelerated, especially under the fast strain rate. At the higher temperature, however, additional concurrent straining under the fast strain rate resulted in a slowing down of the softening kinetics. These behaviors were explained in terms of the strain-induced and/or accelerated polygonization.

INTRODUCTION

Results from several investigations have indicated that the change in properties obtained on annealing a cold-worked metal may be accelerated by concurrent straining during the annealing. A decrease in flow stress, initiated by a "yield drop", has been reported for single crystals which have previously been prestrained at a lower temperature[(1-4)*]. This decrease in stress, which is in addition to the expected "reversible" decrease resulting from a change in temperature, was ascribed to a phenomenon of "work softening"[(1)]. Calorimetric studies of the stored energy remaining after annealing a cold-drawn Au-Ag alloy showed that concurrent straining during annealing greatly accelerated the release of the stored energy. Furthermore, the energy was reduced to a value below that obtained by annealing alone at the given temperature[(5)]. The presence of concurrent straining during annealing of cold-worked polycrystalline pure aluminum specimens at 530°K was to greatly accelerate the rate of softening as indicated by room temperature tensile tests[(6)].

The effect of concurrent stress on the recovery of the flow stress of zinc single crystals previously deformed at a lower temperature in pure shear was investigated by Rinnovatore and Brown[(7)]. It was observed that a concurrent stress less than the instantaneous yield point had no significant effect, while large applied stresses had a marked effect. This suggests that any recovery associated with the application of a concurrent stress is actually due to the concurrent straining which takes place.

A cold-worked state may be completely annealed out by polygonization or recrystallization. The purity[(8-9)], the type and degree of prior deformation, and the annealing temperature[(10-11)], are of importance in determining whether

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*The figures appearing in parentheses pertain to the references appended to this paper.

polygonization or recrystallization would prevail. Polygonization, when studied in relation to concurrent deformation, as in creep, has been of interest primarily toward obtaining a better understanding of the phenomenon associated with deformation per se[12-13]. The fact, however, that polygonization of a cold-worked metal occurs more readily during creep than when annealing under no load would suggest that concurrent straining may induce or accelerate polygonization[13]. If polygonization were the primary softening mechanism, then it would be expected that concurrent straining would accelerate softening by increased polygonization.

Recent creep studies have shown that a cold-worked state may yield inferior creep properties when compared to those obtained from the annealed state[14,16]. It has been suggested that the superimposed creep loading induces recrystallization to occur at a much lower temperature than is normally observed[17]. These observations suggest that concurrent straining may also accelerate recrystallization.

This paper deals with some experimental observations concerned with the effect of concurrent straining, at two different constant strain rates, on the softening kinetics of a cold-rolled vacuum-melted electrolytic iron annealed at temperatures where polygonization and/or recrystallization are found to be important. Throughout this paper the term "static annealing" is used for conventional annealing, i.e., softening without the application of any load. When a load is applied on the specimen resulting in concurrent plastic straining during annealing the term "dynamic annealing" is used.

EXPERIMENTAL PROCEDURE

A vacuum-melted electrolytic iron, provided by Dr. V.F. Zackay of the Ford Motor Company Scientific Laboratory, was used as the test material. A spectographic analysis, made by Mare Island Naval Shipyard, showed, at most, only trace contents of the 16 elements normally checked. The carbon analyzed 0.025 Wt.%. The oxygen and nitrogen contents were given as approximately 0.003 and 0.0001 Wt.%, respectively, by the Ford Motor Scientific Laboratory.

The material was received in the form of 1/4-inch thick by 5-inch wide hot-rolled strips. The strips were pickled and then sheared into 3/4-inch wide pieces. These were reduced 30% in thickness by cold rolling and then annealed at 597°C for 48 hours giving a recrystallized structure. The material was then subjected to a final 60% cold-reduction in preparation for specimens to be used in the annealing studies. Tensile sheet specimens, having a reduced section ¼" in width by 1½" in length, were used for dynamic annealing. Static annealing studies were made with strips, measuring approximately 3/8" x 3/4". A molten carbonate salt bath served as the annealing medium. On reaching the annealing temperature the specimens for dynamic annealing were preloaded to a strain of about 0.02%. Two constant strain rates of 0.005 and 0.6% per hour were used for these studies. All specimens were air cooled from the bath temperature.

The progress of softening was followed by room temperature hardness measurements using the Rockwell 30T Superficial Scale. Frequent microstructural studies were made on a surface which was parallel to the rolling direction and at right angles to the roll diameter. Specimens were etched by several alternate immersions in 65% saturated picric acid in ethyl alcohol and 1% nital. The 60% cold-rolled state had a hardness of 30T-70$\pm$1. Each annealed hardness value reported was obtained from a separate specimen and represents an average of at least three readings.

EXPERIMENTAL RESULTS AND DISCUSSIONS

A. Static Annealing

A survey of the static annealing softening behavior of the 60% cold-rolled metal was made at temperatures ranging from 391 to 597°C. The hardness results are shown in Fig. 1. Microstructures of specimens annealed at different temperatures to approximately the same hardness of Rockwell 30T-52, together with that of the initially cold-rolled state, are shown in Figs. 2 and 3 at magnifications of 150 and 2000, respectively. Partial recrystallization is evident in all the annealed microstructures. The appearance of particles or pits was found to be due to the diamond dust used for polishing.

Two significant observations are illustrated in these photomicrographs. 1. A smaller proportion of recrystallized grains was observed for the same annealed hardness at lower annealing temperatures. A marked change occurred between 482 and 540°C. 2. Somewhat well-defined subgrains were frequently observed in many of the initially elongated grains remaining after annealing at the lower temperatures. This indicates that softening has taken place here by polygonization as well as by recrystallization. Such subgrains were not observed after annealing at the higher temperatures. At these temperatures numerous irregular markings were developed in the elongated grains, as may be seen in Figs. 3A and 3B, suggesting regions of pile up of dislocations such as at deformation bands[18]. In some areas the initiation of subboundaries are indicated.

Referring to Fig. 1, it may be seen that considerable scatter exists only for the hardness data obtained after annealing at the lower temperatures. Such scatter may be considered as suggesting the presence of several strongly competing softening processes. This is supported by the microstructural observations. Thus, above 510°C softening occurs primarily by recrystallization; below 510°C polygonization, in addition to recrystallization, plays an important role.

The presence of competing processes should be reflected in the value of the activation energy. Fig. 4 shows a plot of the logarithmic time interval corresponding to a given hardness drop as a function of the reciprocal of the annealing temperature. Applying the classical Arrhenius equation for activated processes to the data of Fig. 4, two activation energies are obtained. Values of 83,200 and 130,000 calories per mole are obtained for the higher and lower annealing temperatures respectively, with the change in value apparently occurring at about 525°C. The higher activation energy may be interpreted as due to a slowing down of the softening process where both polygonization and recrystallization play important roles. If some polygonization were to take place in a region which would normally undergo recrystallization the strain energy necessary for initiating or continuing recrystallization would be reduced accordingly in such regions. Recrystallization, and therefore softening, is consequently slowed down.

Presumably, at these lower temperatures the interference of recrystallization by polygonization leads to considerable scatter.

The results reported above suggest that some of the strain energy sources for polygonization and recrystallization are, at least initially, the same. Such sources would be primarily at bent lattice regions which are accommodated by excess dislocations of one sign. Calorimetric[19] and electron microscopic[20] studies strongly indicate, however, that the initiation and continuation of these processes differ. If both processes are prevalent, it would be expected that the ensuing competition may lead to erratic annealing behavior and result in scatter, as obtained in the present investigation.

For the temperature-time range investigated, a region was not observed where appreciable softening occurred by polygonization alone. The effect of concurrent straining was therefore studied at 540°C where softening took place essentially by recrystallization and at 482°C where both polygonization and recrystallization occurred for the 60% cold-rolled state. The results are described in the following sections.

B. General Observations on the Effect of Concurrent Straining.

The room temperature hardness values obtained from specimens subjected to dynamic annealing are shown in Fig. 5 as solid lines. The dashed lines represent the static annealing data reproduced from Fig. 1.

Several significant observations may be made when comparing the different annealing curves. 1. Concurrent straining causes an initial hardness drop relative to the static annealing data. This hardness displacement appears to be independent of the strain rates (120 to 1) studied. The static and dynamic annealing curves continue somewhat parallel, especially at the lower temperature, for a significant length of time. 2. The curves for the fast and slow strain-rate tests separate from each other at both temperatures after about the same annealing period of 1½ hours. This time interval corresponds to a strain of about 1% for the fast strain rate. Further

concurrent straining, under the rapid strain rate, causes a relative hardening at the higher temperature while accelerated softening takes place at the lower temperature.

3. The slow strain-rate dynamic annealing data appear to converge gradually with the static annealing data. The maximum strain obtained here corresponds to less than 1%. At the lower temperature the convergence is along the lower values of the scatter band. The observations made above are interpreted in the following sections.

C. Initial Hardness Displacement Caused by Concurrent Straining.

Some small initial strain, probably of the order of the concurrent preload strain of 0.02%, may be sufficient to bring about the sudden hardness displacements shown in Fig. 5. Stokes and Cottrell[1], in referring to the yield drop of "work softening", suggested that a catastrophic reduction in the strain-hardened state takes place. Presumably, a sudden redistribution and self-annihilation of the piled-up dislocations take place as the latter attempt to move away from their barriers under the combination of the elevated temperature and concurrent stress or strain. In the present investigation the relative reduced hardness appears to be independent of temperature, or concurrent strain rate, at least in the early stages. It would appear, however, that the movement of piled-up dislocations away from barriers would depend on the concurrent strain and temperature present. Perhaps some other phenomenon responsible for the hardness difference is eliminated by the initial minimal concurrent strain during annealing. The elimination of solute atmospheres by concurrent straining does not appear plausible at the relatively high temperatures employed. An attempt to explain "work-softening" in aluminum in terms of solute atmospheres was discarded[1].

It has been suggested that the strength of a metal may be increased by the presence of vacancy clusters[21,22]. These clusters may be considered to act as obstacles to dislocation motion in much the same way as precipitates. Experimental evidence indicating the presence of vacancy clusters has been presented using transmission-electron microscopy[23]. Vacancy clusters may form by diffusion of excess vacancies[21,23], the

latter being created during prior plastic deformation[24]. In the present investigation the initial 60% cold-rolling may have created an excess of vacancies. During subsequent heating to the annealing temperature, these vacancies may have agglomerated to form clusters. On concurrent straining such clusters may interact with the moving dislocations, the latter easily acting as a sink for the agglomerated vacancies, resulting in their elimination. Once these are eliminated additional straining would have no further effect, consistent with the experimental results.

The slow strain rate dynamic and static annealing data may be seen to converge with additional annealing time, as shown in Fig. 5, especially at the higher temperature. Any difference associated with the initial hardness drop is thus gradually eliminated. On the assumption that this difference is due to the concentration of vacancy clusters such clusters may then be considered as not affecting the energetics governing recrystallization. An analogy to the elimination of vacancy clusters by recrystallization may be seen in the work reported by Robbins who showed that micropores can be eliminated by the motion of grain boundaries in sintered copper sheet[25]. Cizeron has shown similar effects in the sintering of pure iron[26]. If, however, the softening associated with the initial concurrent strain were due to the redistribution and mutual annihilation of dislocations, then it should be expected that the recrystallization process would have been modified.

D. Convergence and Separation of the Annealing Curves.

The separation of the annealing curves at both temperatures, as shown in Fig. 5, may be explained on the basis of microstructural differences. Up to where the separations occur the microstructures, for all three annealing conditions at each temperature, were essentially identical. Differences, particularly between specimens subjected to the two different concurrent strain rates, were only evident on annealing beyond these separations, and then become continually more apparent with additional annealing time.

Microstructures and corresponding hardness values of specimens annealed for 49

hours at 482°C are shown in Fig. 6. Figs. 6A and 6B represent specimens from the upper and lower regions of the scatter band respectively, as shown in Figs. 1 or 5. Fig. 6C was obtained from the slow strain rate dynamic-annealed specimen. All three microstructures show the presence of subboundaries in the unrecrystallized grains. In the specimen represented by A, however, these boundaries generally formed a lineal rather than the cell-like structure associated with subgrains. In the specimen represented by C the lineal structure was seldom seen; the cell-like structure, although not yet too well formed, was generally present throughout. The specimen represented by B was observed to be intermediate between the above two, although somewhat closer in appearance to the latter one. Under a more rapid concurrent strain rate a well-defined substructure was developed as may be seen in Fig. 6D. A lower hardness, obtained after a given annealing time at a temperature where recrystallization and polygonization are competitive, may then be associated with increased polygonization. The concurrent straining accelerates the polygonization in preference to recrystallization; and thus a more rapid drop in hardness results under the higher concurrent strain rate. This is in contrast to the observations made on static annealing where polygonization, in interrupting the recrystallization process, reduced the rate of softening.

At a temperature of 540°C, where softening during static annealing occurred essentially by recrystallization, the higher concurrent strain rate resulted in a smaller loss of hardness, as shown in Fig. 5. No significant effect was observed as a result of dynamic annealing under the slow strain rate. Microstructures of specimens annealed for 30 hours at 540°C are shown in Fig. 7. Virtually complete recrystallization was obtained for both the static-annealed and slow strain rate dynamic-annealed specimens. Some substructure, however, was occasionally seen in a few grains for the latter specimen. Fig. 7B was taken so as to depict an area containing such grains. Their shape suggests that they were part of elongated grains which may have softened by polygonization. Such polygonization in elongated grains is much more evident in the microstructure,

shown in Fig. 7C, representing the specimen which was subjected to the fast concurrent strain rate. A somewhat coarser, although faint, substructure may also be seen in the recrystallized grains for this specimen.

The separation of the fast strain rate dynamic annealing curve, which results in a slower softening rate and finally leads to a relatively higher annealed hardness, may be thus attributed to two factors. 1. The strain-induced or accelerated polygonization interrupts the recrystallization process such that softening in the unrecrystallized grains continues by polygonization resulting in a relatively lower drop in hardness due to the finer grain structure. 2. The recrystallized grains in turn are hardened by the development of a substructure resulting from bent lattice regions introduced by the concurrent straining as normally takes place during creep.

At the higher temperature where recrystallization is predominant concurrent straining does not appear to affect the rate of recrystallization except by interrupting it with the introduction of another softening process, that of polygonization. At the lower temperature where polygonization is important, during static annealing, concurrent straining accelerates softening by increasing the rate of polygonization. It may be of some significance to note that the point of separation of the data obtained from the fast strain rate specimens, as seen in Fig. 5, occurs at near the same strain of about 1% for both temperatures. The maximum strain attained for the slow strain rate specimens was within this value. It would appear that either an initial small concurrent strain or subsequent strains of greater than 1% are necessary in order to significantly modify the static annealing behavior of the iron used under the conditions studied.

CONCLUSIONS

A. Static Annealing

1. Recrystallization plays a major role in the softening of a 60% cold-rolled iron when annealed at the relatively higher temperatures.

2. At lower annealing temperatures considerable scatter in the room temperature

hardness values is obtained as a result of competition between polygonization and recrystallization. The lower hardness values obtained after a given annealing time is due to increased polygonization. The higher hardness values are associated with an interference of the recrystallization process by the presence of some polygonization. An apparently high value for the activation energy of softening is obtained at the lower temperatures due to such interference.

B. Dynamic Annealing

1. An initial hardness drop below that observed for static annealing is obtained as a result of concurrent straining during the annealing. This drop appears to be independent of strain rate or temperature. The interaction of vacancy clusters with strain-induced moving dislocations may account for such a hardness drop.

2. Concurrent straining at a temperature where polygonization as well as recrystallization is important accelerates the rate of softening. This results due to an increase in polygonization.

3. A fast strain rate during dynamic annealing at a temperature where recrystallization plays a major role during static annealing, results in a slowing down of the softening process. The concurrent straining interrupts the recrystallization by the inducement and/or acceleration of polygonization.

4. Either a minimal initial strain or a strain of greater than about 1% is necessary for modification of the static annealing curves by the presence of concurrent straining.

ACKNOWLEDGMENTS

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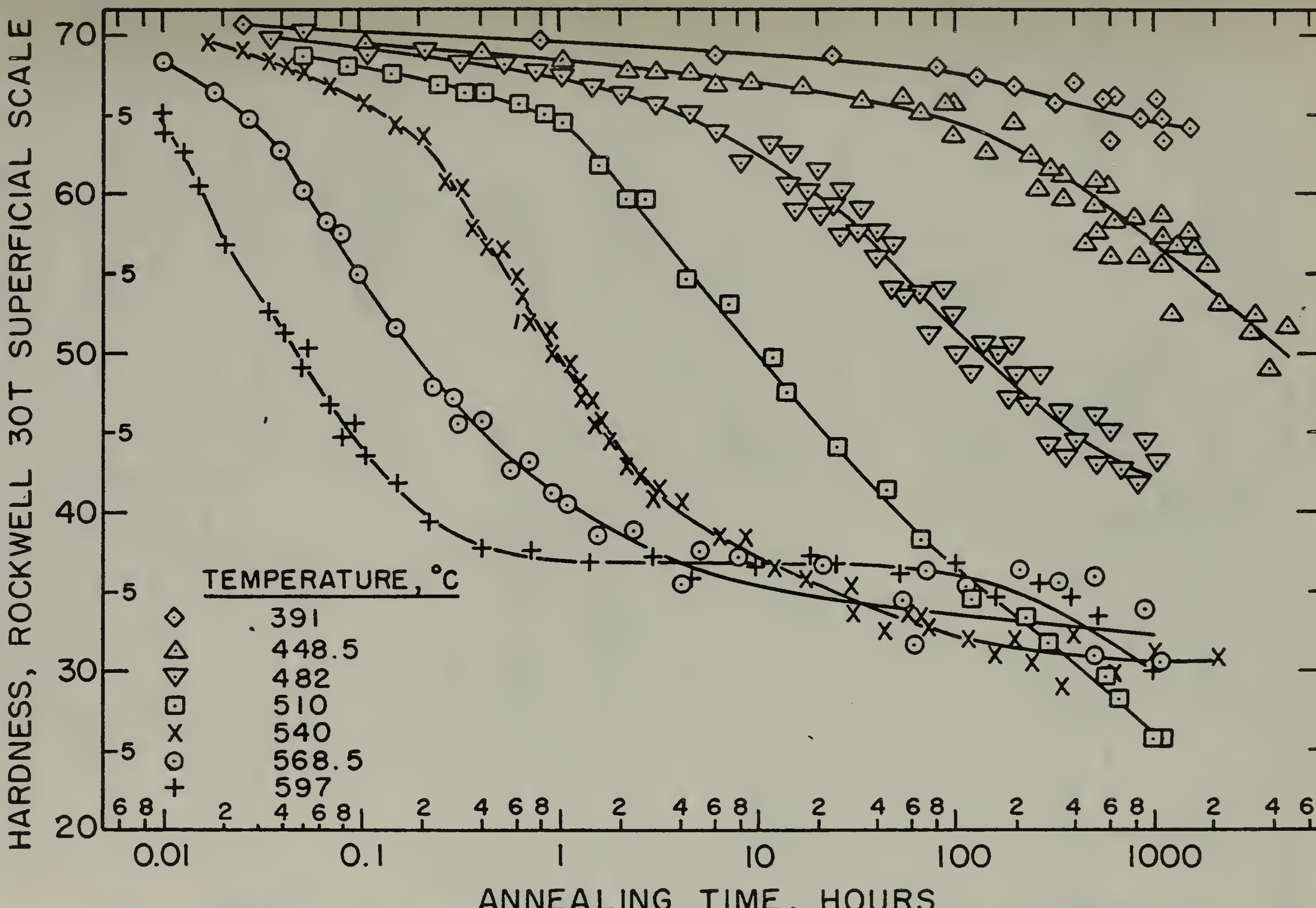


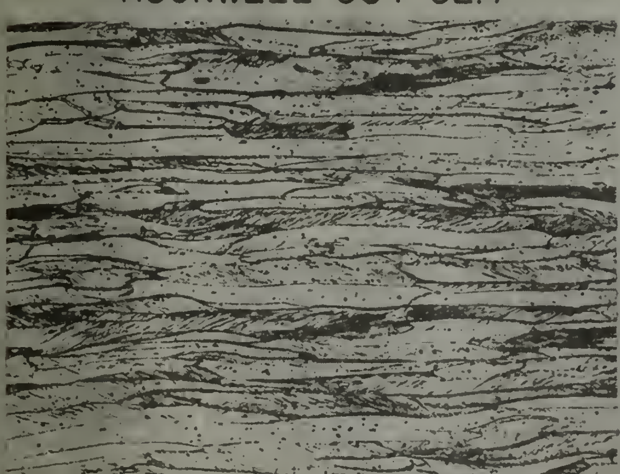
Fig. 1 The effect of annealing time at several temperatures on the room temperature hardness for a 60% cold-rolled vacuum-melted iron.

A. 597°C, 0.033 HOURS
ROCKWELL 30T-52.6

B. 540°C, 0.70 HOURS
ROCKWELL 30T-51.9

C. 482°C, 96.0 HOURS
ROCKWELL 30T-52.4

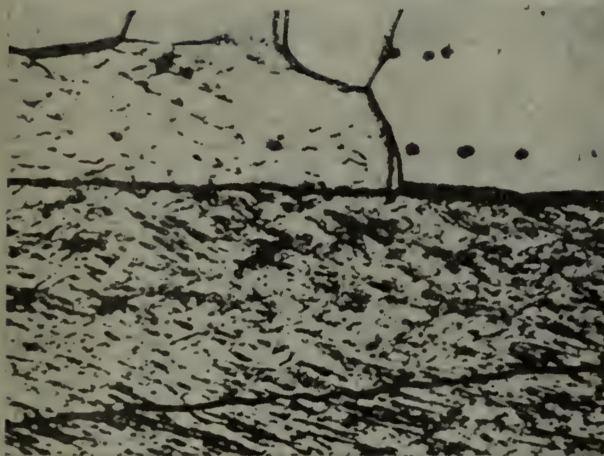
D. 448.5°C, 1242 HOURS
ROCKWELL 30T-52.5



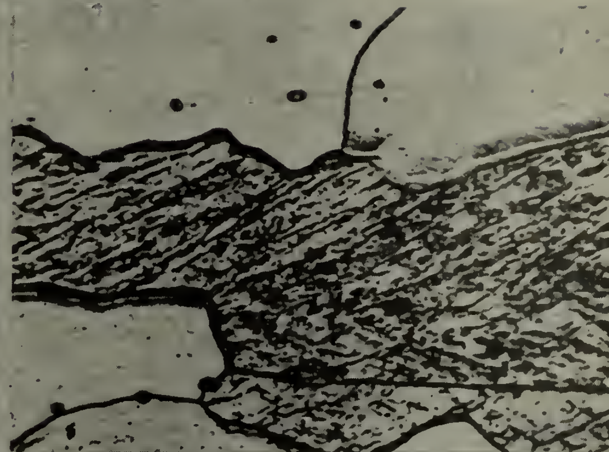
E. AS 60% COLD-ROLLED
ROCKWELL 30T-71

Fig. 2 Microstructures of an initially 60% cold-rolled vacuum-melted iron following annealing at different temperatures to approximately the same hardness. A decrease in the number of recrystallized grains with lower annealing temperatures is depicted. (150X)

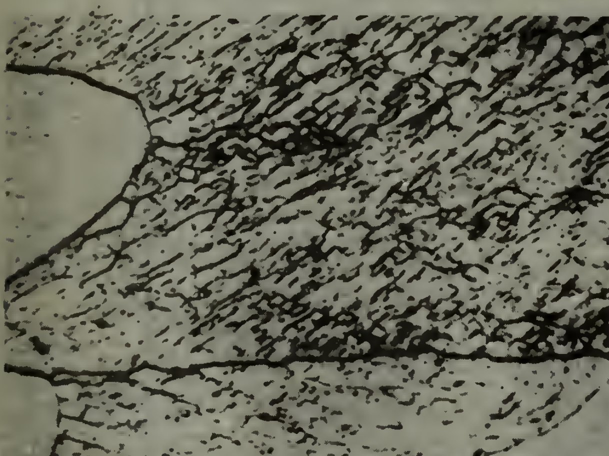
—— ROLLING DIRECTION ⟶



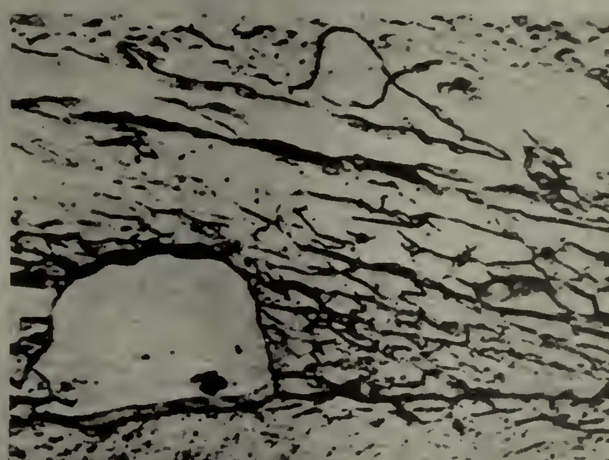
A. 597 °C, 0.033 HOURS
ROCKWELL 30T-52.6



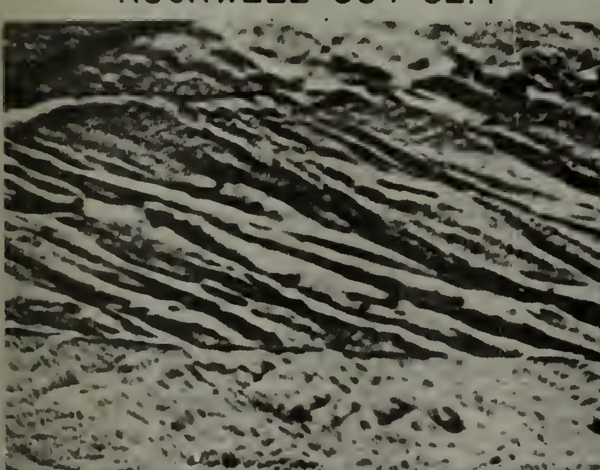
B. 540°C, 0.70 HOURS
ROCKWELL 30T-51.9



C. 482 °C, 96.0 HOURS
ROCKWELL 30T-52.4



D. 448.5°C, 1242 HOURS
ROCKWELL 30T-52.5



E. AS 60 % COLD-ROLLED
ROCKWELL 30T-71

Fig. 3 Microstructures of specimens used for Fig. 2 taken at a higher magnification. Details of the structure within the previously elongated grains resulting from annealing at different temperatures are depicted. (2000X)

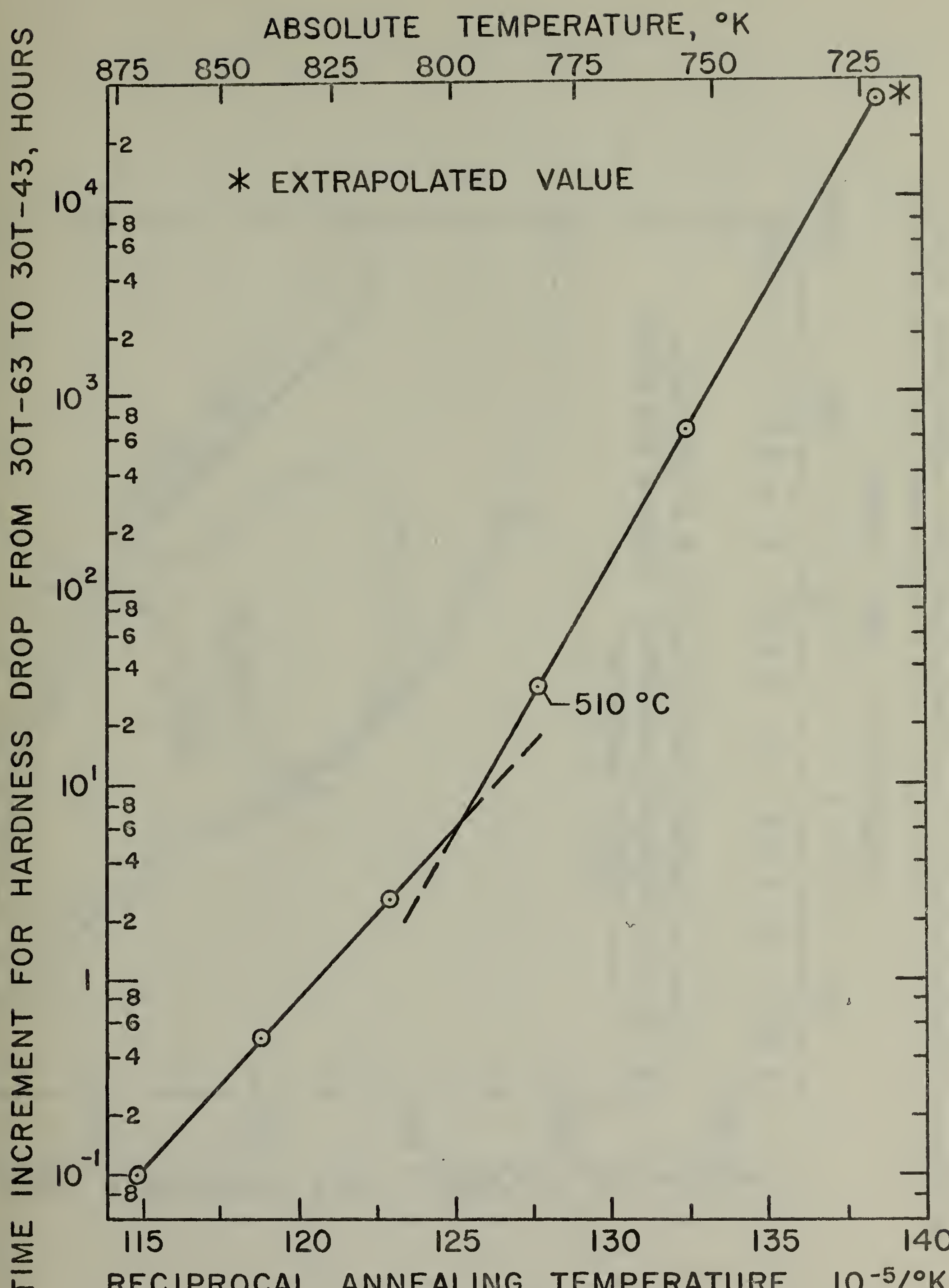


Fig. 4 Reciprocal annealing temperature-time curve used for the determination of activation energy for a 60% cold-rolled vacuum-melted iron. Time refers to the interval for a drop in hardness from Rockwell 30T-63 to 30T-43 obtained from the data shown in Fig. 1.

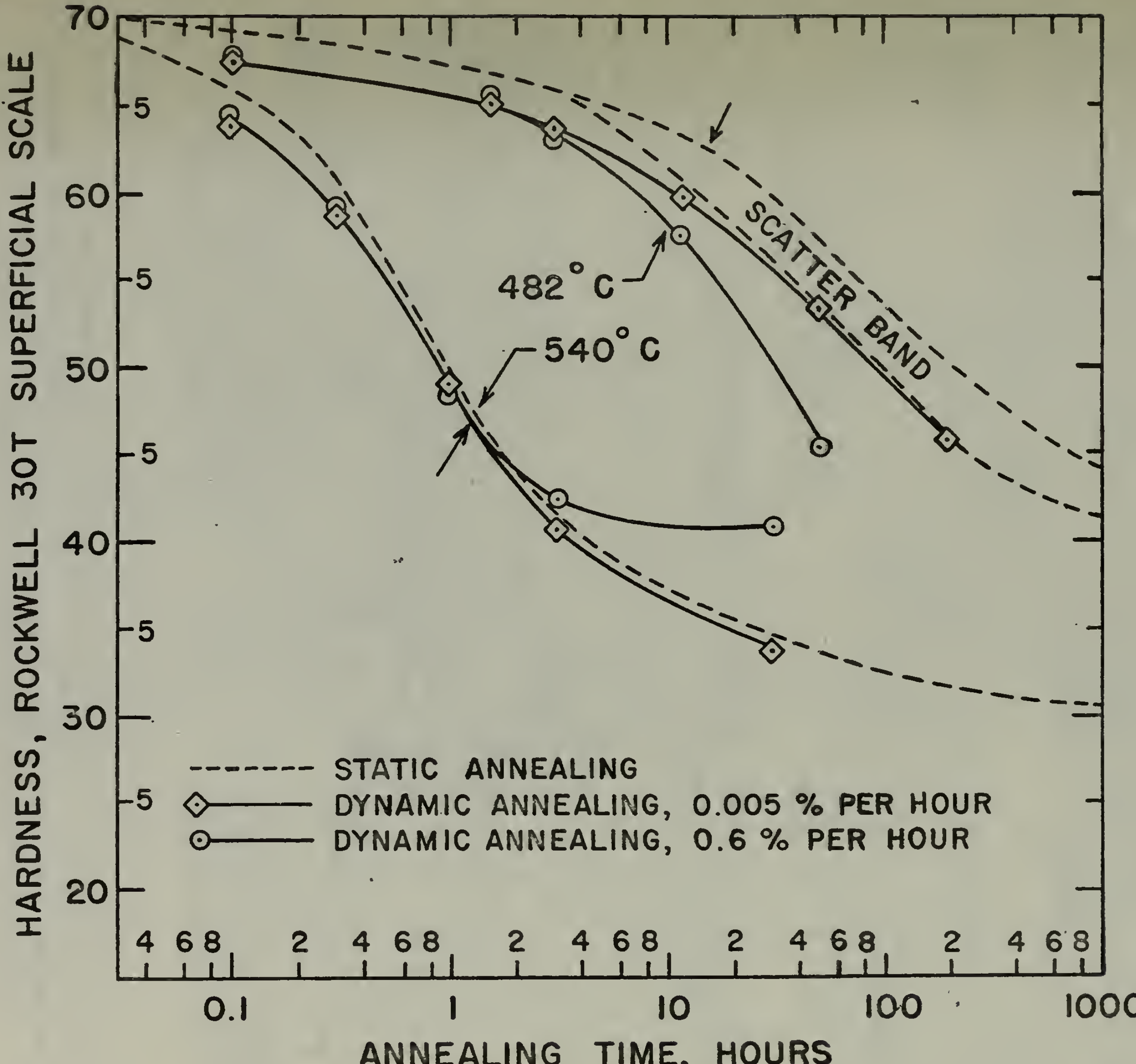


Fig. 5 The effect of concurrent straining on the softening behavior for a 60% cold-rolled vacuum-melted iron, at two temperatures where polygonization at 482°C and recrystallization at 540°C predominate during static annealing.

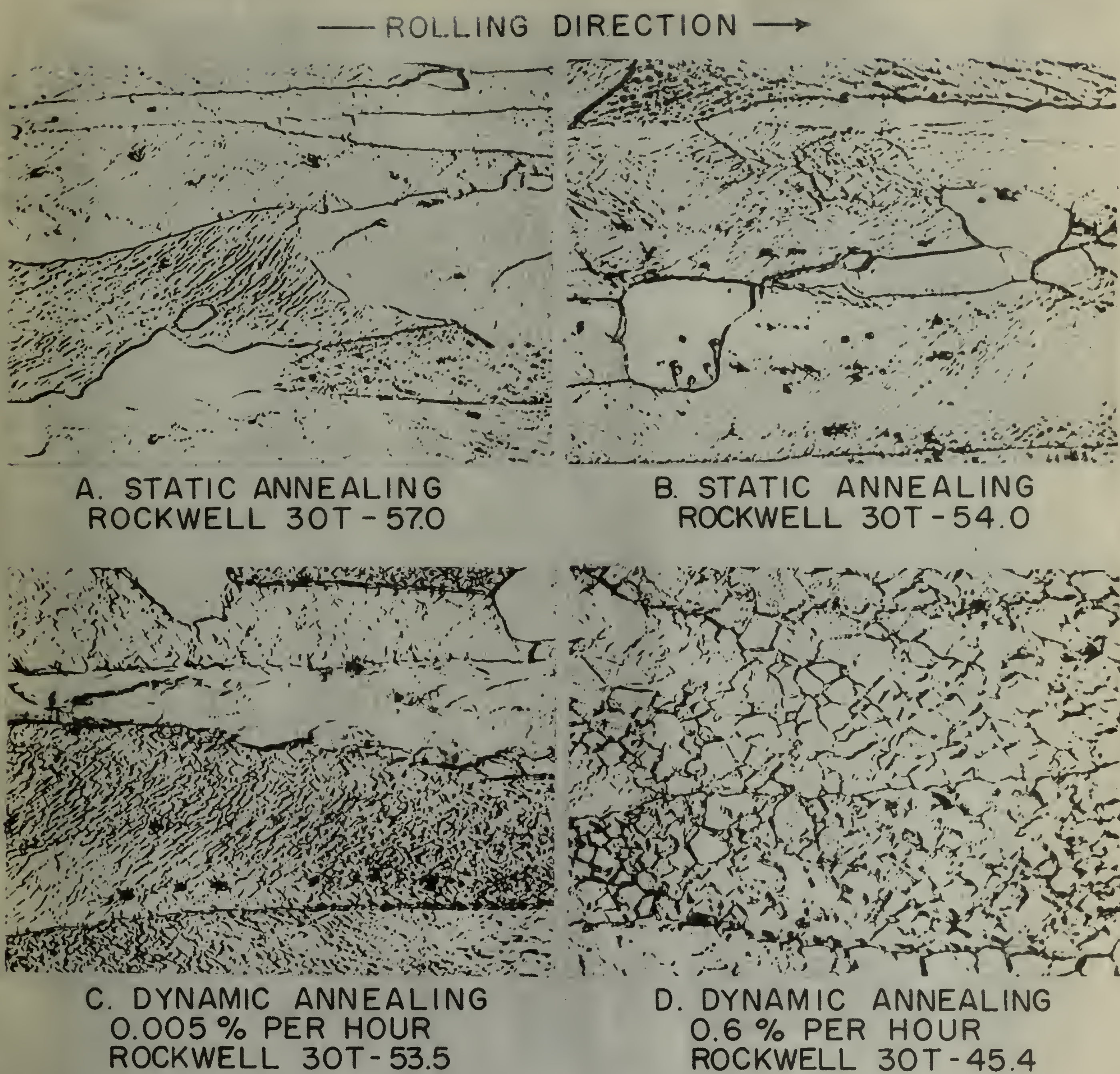
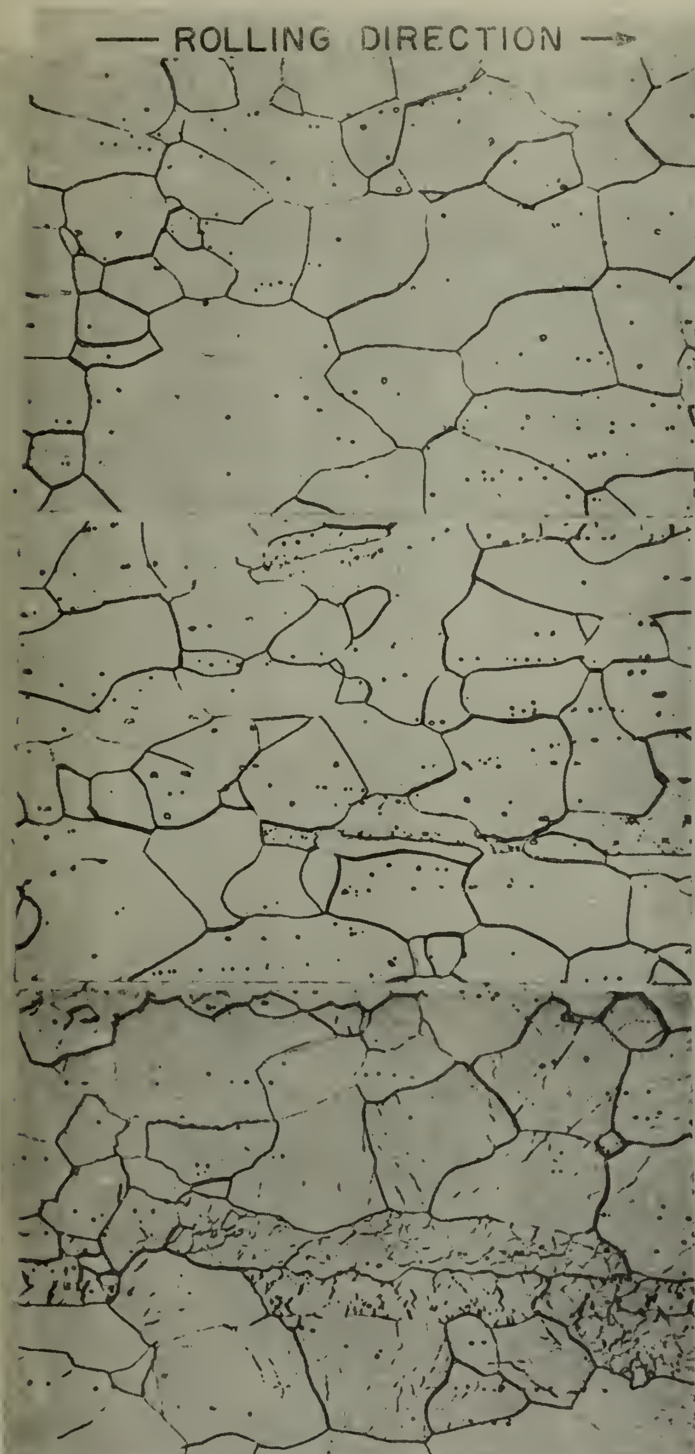


Fig. 6 Microstructures of specimens of an initially 60% cold-rolled vacuum-melted iron annealed at 482°C for 49 hours. The nature and development of the substructure and its relation to the hardness obtained during both similar and different annealing treatments are indicated. (1000X)



A.
STATIC ANNEALING
ROCKWELL 30T-33.7

B.
DYNAMIC ANNEALING
0.005 % PER HOUR
ROCKWELL 30T-33.8

C.
DYNAMIC ANNEALING
0.6 % PER HOUR
ROCKWELL 30T-40.7

Fig. 7 Microstructures of specimens of an initally 60% cold-rolled vacuum-melted iron annealed at 540°C for 30 hours. The development of a substructure in both the originally elongated grains and new recrystallized grains as a result of concurrent straining is depicted. (500X)